

Volume 144, 1999

# JOURNAL OF SOLID STATE CHEMISTRY

Editors

J. M. HONIG    M. G. KANATZIDIS

Associate Editor

H.-C. ZUR LOYE



ACADEMIC PRESS

Copyright © 1999 by Academic Press  
All Rights Reserved

No part of this publication may be reproduced or transmitted in any form or by any means, electronic or mechanical, including photocopying, recording, or any information storage and retrieval system, without permission in writing from the Publisher. *Exceptions:* It is the policy of Academic Press that authors need not obtain permission in the following cases only: (1) to use their original figures or tables in their future works; (2) to make copies of their papers for use in their classroom teaching; and (3) to include their papers as part of their dissertations.

The appearance of the code at the bottom of the first page of an article in this journal indicates the Publisher's consent that copies of the article may be made for personal or internal use, or for the personal or internal use of specific clients. This consent is given on the condition, however, that the copier pay the stated per copy fee through the Copyright Clearance Center, Inc. (222 Rosewood Drive, Danvers, Massachusetts 01923), for copying beyond that permitted by Sections 107 or 108 of the U.S. Copyright Law. This consent does not extend to other kinds of copying, such as copying for general distribution, for advertising or promotional purposes, for creating new collective works, or for resale. Copy fees for pre-1999 articles are as shown on the article title pages; if no fee code appears on the title page, the copy fee is the same as for current articles.

0022-4596/99 \$30.00

MADE IN THE UNITED STATES OF AMERICA

This journal is printed on acid-free paper.



# Contents of Volume 144

Number 1, April 1999

## LETTER TO THE EDITOR

|                                                                     |                       |     |
|---------------------------------------------------------------------|-----------------------|-----|
| Giant Oxygen Isotope Effect in Charge-Ordered Manganese Perovskites | R. Mahesh and M. Itoh | 232 |
|---------------------------------------------------------------------|-----------------------|-----|

## REGULAR ARTICLES

|                                                                                                                                                                                                                                                |                                                                                                                                                                                              |     |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|
| Properties and Reactivity of Mechanochemically Synthesized Tin Sulfides                                                                                                                                                                        | P. Baláž, T. Ohtani, Z. Bastl, and E. Boldižárová                                                                                                                                            | 1   |
| Electrochemical Investigation of Oxygen Intercalation into $\text{La}_2\text{CuO}_{4+\delta}$ Phases                                                                                                                                           | E. Magnone, G. Cerisola, M. Ferretti, and A. Barbucci                                                                                                                                        | 8   |
| Rietveld Refinement of the Crystal Structure of Cs(IV), a <i>d</i> -Electron Metal                                                                                                                                                             | J. L. Delattre and J. V. Badding                                                                                                                                                             | 16  |
| Electron Paramagnetic Resonance Spectrum of $\text{Pr}^{4+}$ in $\text{Sr}_2\text{CeO}_4$                                                                                                                                                      | Yukio Hinatsu, Makoto Wakeshima, Norman Edelstein, and Ian Craig                                                                                                                             | 20  |
| Characterization of Various Magnesium Oxides by XRD and $^1\text{H}$ MASNMR Spectroscopy                                                                                                                                                       | Maria Angeles Aramendia, José Antonio Benitez, Victoriano Borau, César Jiménez, José María Marinas, José Rafael Ruiz, and Francisco Urbano                                                   | 25  |
| Crystal and Electronic Structures and Linear Optics of Strontium Pyroborate                                                                                                                                                                    | Q.-S. Lin, W.-D. Cheng, J.-T. Chen, and J.-S. Huang                                                                                                                                          | 30  |
| Crystal Structure of Sodium Rare Earth Oxyborates $\text{Na}_2\text{Ln}_2(\text{BO}_3)_2\text{O}$ ( $\text{Ln} = \text{Sm}, \text{Eu}, \text{and Gd}$ ) and Optical Analysis of $\text{Na}_2\text{Gd}_2(\text{BO}_3)_2\text{O}:\text{Eu}^{3+}$ | G. Corbel, M. Leblanc, E. Antic-Fidancev, and M. Lemaître-Blaise                                                                                                                             | 35  |
| Acidic and Hydrophobic Microporous Clays Pillared with Mixed Metal Oxide Nano-Sols                                                                                                                                                             | Yang-Su Han, Shoji Yamanaka, and Jin-Ho Choy                                                                                                                                                 | 45  |
| Synthèse et structure cristalline de $\text{Na}_3\text{NbO}(\text{AsO}_4)_2$                                                                                                                                                                   | Khaled Hizaoui, Noureddine Jouini, et Tahar Jouini                                                                                                                                           | 53  |
| Structure of $\text{TiSr}_2\text{NiO}_{4+\delta}$ by High-Resolution Neutron Powder Diffraction                                                                                                                                                | C. S. Knee and M. T. Weller                                                                                                                                                                  | 62  |
| Potassium Lanthanum Nitrate System: Phase Equilibria and Thermal and Structural Properties                                                                                                                                                     | Anne-Elisabeth Gobichon, Jean-Paul Auffrédic, and Daniel Louër                                                                                                                               | 68  |
| A New Series of Complex Perovskites $\text{La}_{1-x}\text{Sr}_x\text{Cr}_{1-x}\text{Ti}_x\text{O}_3$ : Structural Characterization                                                                                                             | Roger H. Mitchell and Anton R. Chakhmouradian                                                                                                                                                | 81  |
| Application of a Modified Ultrasonic Aerosol Device to the Synthesis of $\text{SnO}_2$ and $\text{Pt/SnO}_2$ for Gas Sensors                                                                                                                   | M. V. Cabañas, G. Delabouglise, M. Labeau, and M. Vallet-Regí                                                                                                                                | 86  |
| Phase Relations in the System $\text{In}_2\text{O}_3\text{--TiO}_2\text{--Fe}_2\text{O}_3$ at $1100^\circ\text{C}$ in Air                                                                                                                      | Francisco Brown, Maria J. R. Flores, Noboru Kimizuka, Yuichi Michiue, Mitsuko Onoda, Takahiko Mohri, Masaki Nakamura, and Nobuo Ishizawa                                                     | 91  |
| Defect and Ordered Tungsten Oxides Encapsulated Inside $2\text{H--WX}_2$ ( $\text{X} = \text{S}$ and $\text{Se}$ ) Fullerene-Related Structures                                                                                                | Jeremy Sloan, John L. Hutchison, Reshef Tenne, Yishay Feldman, Tatyana Tsirlina, and Moshe Homyonfer                                                                                         | 100 |
| Gibbs Free Energy of Formation of $\text{LnBa}_2\text{Cu}_3\text{O}_{7-x}$ Phases Determined by the EMF Method ( $\text{Ln} = \text{Yb}, \text{Tm}, \text{Er}, \text{Ho}, \text{Dy}$ )                                                         | M. Kopyto and K. Fitzner                                                                                                                                                                     | 118 |
| $\text{Sr}_4\text{Ru}_{3.05}\text{O}_{12}$ : A New Member of the Hexagonal Perovskite Family                                                                                                                                                   | C. Renard, S. Daviero-Minaud, M. Huvé, and F. Abraham                                                                                                                                        | 125 |
| Preparation of $\alpha\text{-MnO}_2$ with an Open Tunnel                                                                                                                                                                                       | Yuji Muraoka, Hiroshi Chiba, Toshiyuki Atou, Masae Kikuchi, Kenji Hiraga, Yasuhiko Syono, Shoko Sugiyama, Sadaaki Yamamoto, and Jean-Calude Grenier                                          | 136 |
| Concomitant Intercalation and Decomplexation of Ferrocene Sulfonates in Layered Double Hydroxides                                                                                                                                              | Sandrine Morlat-Thérias, Christine Mousty, Pierre Palvadeau, Philippe Molinié, Philippe Léone, Jean Rouxel, Christine Taviot-Guého, Abderrahman Ennaoui, André de Roy, and Jean Pierre Besse | 143 |

|                                                                                                                                                                                     |                                                                                            |     |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------|-----|
| <b>Crystal Structures of <math>U(Ta,W)_5O_{16}</math> and <math>U(Ta,W)_2O_8</math> Revealed by High-Resolution Transmission Electron Microscopy</b>                                | Charlotta Askeljung and Margareta Sundberg                                                 | 152 |
| <b>Effect of Polymer Matrix on the Phase Transitions of <math>CuFe_2O_4</math></b>                                                                                                  | S. Roy and J. Ghose                                                                        | 159 |
| <b>Structure et propriétés magnétiques du composé <math>NH_4Fe_3(H_2PO_4)_6(HPO_4)_2 \cdot 4H_2O</math></b>                                                                         | A. Mgaidi, H. Boughzala, A. Driss, R. Clerac, et C. Coulon                                 | 163 |
| <b>Structure Characterization and Magnetic Behavior of <math>NaNi_4(PO_4)_3</math> and <math>KMn_4(PO_4)_3</math></b>                                                               | Abdelaali Daidouh, J. L. Martinez, C. Pico, and M. L. Veiga                                | 169 |
| <b>Lanthanum and Praseodymium Bromide Pnictides. A Convergence of Interstitial Chemistry in Cluster Halides and Intermetallic Pnictides</b>                                         | Elizabeth A. Jensen, Laura M. Hoistad, and John D. Corbett                                 | 175 |
| <b>Crystal Structures and Lattice Distortions of <math>\sigma</math>-Type Layered Vanadium Bronzes: <math>\sigma-M_{0.25}V_2O_5 \cdot H_2O</math> (<math>M = Mg, Co, Ni</math>)</b> | Yoshio Oka, Takeshi Yao, and Naoichi Yamamoto                                              | 181 |
| <b>Calorimetric Investigation of Tricritical Behavior in Tetragonal <math>Pb(Zr_xTi_{1-x})O_3</math></b>                                                                            | George A. Rossetti, Jr. and Alexandra Navrotsky                                            | 188 |
| <b>Polymorphism in <math>Bi_5Pb_3O_{10.5}</math></b>                                                                                                                                | Akiteru Watanabe, Yoshizo Kitami, Satoshi Takenouchi, Jean-Claude Boivin, and Nigel Sammes | 195 |
| <b>Crystal Structure of the <math>(K_{0.87}Bi_{0.13})BiO_3</math> Superconductor</b>                                                                                                | N. R. Khasanova, F. Izumi, T. Kamiyama, K. Yoshida, A. Yamamoto, and S. Tajima             | 205 |
| <b>The High-Temperature Phases of <math>WO_3</math></b>                                                                                                                             | Tom Vogt, Patrick M. Woodward, and Brett A. Hunter                                         | 209 |
| <b>BRIEF COMMUNICATIONS</b>                                                                                                                                                         |                                                                                            |     |
| <b>Magnetic Properties of Ruthenium Pyrochlores <math>Y_2Ru_2O_7</math> and <math>Lu_2Ru_2O_7</math></b>                                                                            | Nobuyuki Taira, Makoto Wakeshima, and Yukio Hinatsu                                        | 216 |
| <b>Supercapacitor Behavior with KCl Electrolyte</b>                                                                                                                                 | Hee Y. Lee and J. B. Goodenough                                                            | 220 |
| <b>Large Intragrain Magnetoresistance above Room Temperature in the Double Pervoskite <math>Ba_2FeMoO_6</math></b>                                                                  | A. Maignan, B. Raveau, C. Martin, and M. Hervieu                                           | 224 |
| <b><math>Sr_3MO_4F</math> (<math>M = Al, Ga</math>)—A New Family of Ordered Oxyfluorides</b>                                                                                        | T. Vogt, P. M. Woodward, B. A. Hunter, A. K. Prodjosantoso, and B. J. Kennedy              | 228 |

## Number 2, May 1999

### REGULAR ARTICLES

|                                                                                                                                                               |                                                                                                                               |     |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------|-----|
| <b>Synthesis of Nanocrystalline Iron Monoarsenide via a Reductive Recombination Pathway</b>                                                                   | X. M. Zhang, C. Wang, X. F. Qian, Y. Xie, and Y. T. Qian                                                                      | 237 |
| <b>Ordering of <math>Ce^{III}/Ce^{IV}</math> and Interstitial Oxygens in <math>CeTaO_{4+x}</math> (<math>x \approx 0.17</math>) Superstructure</b>            | John G. Thompson, A. David Rae, Nina Bliznyuk, and Ray L. Withers                                                             | 240 |
| <b>Equilibre solide—liquide dans le système ternaire <math>H_2O-Na_2SO_4-Na_2HPO_4</math>: Etude de quelques isothermes entre 0 et 33°C</b>                   | E. Bel Madani, M. Nadifiyine, A. Mokhlisse, et B. Tanouti                                                                     | 247 |
| <b>An Investigation of the Europium Valence in <math>EuCu_{1.75}P_2</math> by Eu Mössbauer and Eu <math>L_{III}</math>-Edge X-Ray Absorption Spectroscopy</b> | Robert M. Bornick, Angelica M. Stacy, R. Dean Taylor, and George H. Kwei                                                      | 252 |
| <b>Structural Properties and Thermal Stability of <math>Bi_8Pb_5O_{17}</math> Fast Ion Conducting Phases</b>                                                  | Monica Santarosa, Lara Righi, Mauro Gemmi, Adolfo Speghini, Andrea Migliori, Gianluca Calestani, and Marco Bettinelli         | 255 |
| <b>Intermolecular Bonds Bridging Two Anthracene Molecules in <math>\alpha</math>-Cyclodextrin</b>                                                             | Rodion Belosludov, Tohru Hiwada, Yoshiyuki Kawazoe, Kaoru Ohno, Takehisa Yoshinari, Akimasa Ohnishi, and Shin-ichiro Nagasaka | 263 |
| <b>Preparation and Structure Refinement of Monoclinic Hydroxyapatite</b>                                                                                      | Toshiyuki Ikoma, Atsushi Yamazaki, Satoshi Nakamura, and Masaru Akao                                                          | 272 |

|                                                                                                                                                                                                                                                                                               |                                                                                                                   |     |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------|-----|
| <b>Crystal Chemistry of Defective Intermetallics with Empty Triangular Metalloid Channels: The New Phosphide <math>\text{Zr}_6\text{Cr}_{60}\text{P}_{39}</math></b>                                                                                                                          | C. Le Sénéchal, V. Babizhetskyy, S. Députier, J. Y. Pivan, and R. Guérin                                          | 277 |
| <b>Synthesis of Two New Metallic Alkali Metal Silver Selenides, <math>\text{K}_2\text{Ag}_{12}\text{Se}_{7.11}</math> and <math>\text{RbAg}_5\text{Se}_3</math>, from Supercritical Ethylenediamine</b>                                                                                       | Mehtap Emirdag, George L. Schimek, William T. Pennington, and Joseph W. Kolis                                     | 287 |
| <b>Mo(V) Phosphates with an Opened Framework: <math>\text{Li}_{4-x}\text{A}_x^+(\text{MoO})_3(\text{PO}_4)_3\text{P}_2\text{O}_7</math> (<math>\text{A} = \text{Na, Ag, Cs, K}</math>), <math>\text{Li}_{4-x}\text{Ba}_{x/2}^{2+}(\text{MoO})_3(\text{PO}_4)_3\text{P}_2\text{O}_7</math></b> | S. Ledain, A. Leclaire, M. M. Borel, M. Hervieu, J. Provost, and B. Raveau                                        | 297 |
| <b>Crystal and Magnetic Structure of New Ternary Uranium Intermetallics: <math>\text{U}_3\text{TiX}_5</math> (<math>\text{X} = \text{Ge, Sn}</math>)</b>                                                                                                                                      | P. Boulet, G. M. Gross, G. André, F. Bourée, and H. Noël                                                          | 311 |
| <b>Characterization of a New Organic-Cation Cyclotetraphosphate: <math>(1,4\text{-HOC}_6\text{H}_4\text{NH}_3)_4\text{P}_4\text{O}_{12} \cdot 6\text{H}_2\text{O}</math></b>                                                                                                                  | E. H. Soumhi, I. Saadoune, A. Driss, and T. Jouini                                                                | 318 |
| <b>Structures of Two New Polymorphic Forms of Hexavalent Tungsten Oxide Phosphates</b>                                                                                                                                                                                                        | Masafumi Hanawa and Hideo Imoto                                                                                   | 325 |
| <b>Differences and Similarities between the Isotypic Antimonides <math>\text{MFe}_{1-x}\text{Sb}</math>, <math>\text{ScCo}_{1-x}\text{Sb}</math>, and <math>\text{MNiSb}</math> (<math>\text{M} = \text{Zr, Hf}</math>)</b>                                                                   | Holger Kleinke and Claudia Felser                                                                                 | 330 |
| <b>Structural Considerations and Polarization Energy of Some <math>\text{PbFCl}</math>-Type Compounds</b>                                                                                                                                                                                     | M. Sieskind and J. Morel                                                                                          | 339 |
| <b>Copper Precursor Effect on Reducibility and Titania Phases Concentration of Sol-Gel <math>\text{Cu/TiO}_2</math> Catalyst</b>                                                                                                                                                              | X. Bokhimi, O. Novaro, R. D. Gonzalez, T. López, O. Chimal, A. Asomoza, and R. Gómez                              | 349 |
| <b>Dependence of ac Conductivity on Temperature and Frequency in Cerium(III) Nitrate Crystal</b>                                                                                                                                                                                              | Riki Kawashima, Hironori Takahashi, and Hiroshi Isoda                                                             | 354 |
| <b><i>In Situ</i> Structural Study of 4V-Range Lithium Extraction/Insertion in Fluorine-Substituted <math>\text{LiMn}_2\text{O}_4</math></b>                                                                                                                                                  | M. R. Palacin, F. Le Cras, L. Seguin, M. Anne, Y. Chabre, J. M. Tarascon, G. Amatucci, G. Vaughan, and P. Strobel | 361 |
| <b>A Powder Neutron Diffraction Study of the Magnetic Structure of <math>\text{FeV}_2\text{S}_4</math></b>                                                                                                                                                                                    | Anthony V. Powell, Clemens Ritter, and Paz Vaqueiro                                                               | 372 |
| <b>Structural Study of Modified <math>\text{Bi}_4\text{V}_2\text{O}_{10+\delta}</math> Phase (<math>\delta = 0, 0.5, 1</math>): Influence of Antimony Contribution</b>                                                                                                                        | S. Sorokina, R. Enjalbert, P. Baules, A. Castro, and J. Galy                                                      | 379 |
| <b>Room Temperature Solid State Synthesis and Characterization of a New Chromium Thiophosphate <math>\text{Cr}_4(\text{P}_2\text{S}_6)_3</math></b>                                                                                                                                           | Zhong-Le Huang, Jing-Tai Zhao, Jin-Xiao Mi, Shao-Yu Mao, and Lan-Sum Zheng                                        | 388 |
| <b>Some Features of the Reduction Process from <math>\text{CrVO}_4</math> to <math>\text{CrVO}_3</math></b>                                                                                                                                                                                   | Irma L. Botto, Marta B. Vassallo, Manlio Occhiuzzi, Dante Cordischi, and Piero Porta                              | 392 |
| <b>Partial Oxygen Ordering in Cubic Perovskite <math>\text{REBa}_2\text{Fe}_3\text{O}_{8+w}</math> (<math>\text{RE} = \text{Gd, Eu, Sm, Nd}</math>)</b>                                                                                                                                       | J. Lindén, P. Karen, A. Kjekshus, J. Miettinen, and M. Karppinen                                                  | 398 |
| <b>A New Layered Bismuthate (<math>\text{Sr,K})_3\text{Bi}_2\text{O}_7</math>: Synthesis and Crystal Structure</b>                                                                                                                                                                            | J. S. Pshirkov, S. M. Kazakov, C. Bougerol-Chaillout, P. Bordet, J. J. Capponi, S. N. Putilin, and E. V. Antipov  | 405 |
| <b>Synthesis and Structure of a New Ternary Silver-Rich Sulfide <math>\text{BaAg}_8\text{S}_5</math></b>                                                                                                                                                                                      | Catherine E. Check, Chong Zheng, Jianhua Zhang, and Bogdan Dabrowski                                              | 409 |
| <b>Compressibility and Vibrational Modes in Solid <math>\text{As}_4\text{O}_6</math></b>                                                                                                                                                                                                      | Andrzej Grzechnik                                                                                                 | 416 |
| <b>Point Defect Energies for Strontium Titanate: A Pair-Potentials Study</b>                                                                                                                                                                                                                  | Jill Crawford and Patrick Jacobs                                                                                  | 423 |
| <b>Electron Diffraction Study of Intercalation Compounds Derived from <math>1\text{T-MoS}_2</math></b>                                                                                                                                                                                        | F. Wypych, C. Solenthaler, R. Prins, and Th. Weber                                                                | 430 |
| <b>Preparation and Characterization of Garnet Phosphor Nanoparticles Derived from Oxalate Coprecipitation</b>                                                                                                                                                                                 | Teng-Ming Chen, S. C. Chen, and Chao-Jung Yu                                                                      | 437 |
| <b>The First Vanadium Gallophosphates: Hydrothermal Synthesis and Structure of <math>\text{Rb}[(\text{VO})(\text{H}_2\text{O})\text{Ga}(\text{PO}_4)_2]</math> and <math>\text{Cs}[(\text{VO})(\text{H}_2\text{O})\text{Ga}(\text{PO}_4)_2]</math></b>                                        | Robert P. Hammond and Jon A. Zubieta                                                                              | 442 |

|                                                                                                                                                       |                                                                           |     |
|-------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------|-----|
| <b>Yb: Borate Glass with High Emission Cross Section</b>                                                                                              | Chun Jiang, Fuxi Gan, Junzhou Zhang, Peizhen Deng, and Guosong Huang      | 449 |
| <b>Charge Density Wave Transitions Induced in <math>\text{Nb}_3\text{Se}_4</math> and <math>\text{Nb}_3\text{S}_4</math> by Indium Intercalation</b>  | F. W. Boswell, J. C. Bennett, and A. Prodan                               | 454 |
| <b>Effective Oxygen Content and Properties of <math>\text{La}_{0.74}\text{Ca}_{0.26}\text{MnO}_{3+d}</math> as a Function of Synthesis Conditions</b> | B. Dabrowski, P. W. Klamut, Z. Bukowski, R. Dybzinski, and J. E. Siewenie | 461 |
| <b>BRIEF COMMUNICATION</b>                                                                                                                            |                                                                           |     |
| <b>Temperature-Dependent Structural Behavior of <math>\text{Bi}_{0.5}\text{Nd}_{1.5}\text{Ru}_2\text{O}_7</math></b>                                  | M. D. Field, B. J. Kennedy, B. A. Hunter, and T. Vogt                     | 467 |
| <b>AUTHOR INDEX FOR VOLUME 144</b>                                                                                                                    |                                                                           | 470 |

## NOTICE

The Subject Index for Volume 144 will appear in the December 1999 issue as part of a cumulative index for the year 1999.